

invention. In a first operation, the population of carbon nanotubes is dispersed in a liquid vehicle. That is, it is preferred to attain a state in which the various nanotubes are individually dispersed in the liquid vehicle so as to minimize any bundling or agglomerating of the nanotubes that may otherwise occur. This operation or series of operations is designated as **110** in FIG. 2. A preferred technique for attaining such highly dispersed state is to form an aqueous dispersion of the nanotubes by sonicating the liquid. One or more surfactants or other additives may be added to the liquid to promote solubilizing the nanotubes. After sufficient dispersion, impurities are then removed, such as by centrifuging.

[0057] Operation **120** in FIG. 2 illustrates selection of one or more suitable density adjusting agents for use in the length separation process described herein. Preferably, the density adjusting agent is used to form a liquid, i.e. the race layer, having a density such that the difference in the densities of the various length nanotubes to be separated is relatively small as compared to the difference between the average density of the dispersed nanotubes and the density of the race layer liquid. Specifically, in selecting a density adjusting agent, the agent is preferably selected and combined with one or more other liquids to form the race layer(s) such that the density of the race layer satisfies the following relationship. The density of the race layer liquid is preferably such that the difference between (i) the density of the race layer liquid and (ii) the average density of the carbon nanotubes in the dispersed sample is greater, and preferably significantly greater, than the difference between (ii) and (iii) the density of any species of carbon nanotubes in the dispersed sample. As explained herein, for length separation of SWCNTs in an aqueous dispersion, iodixanol has been found to be a preferred density adjusting agent. However, the present invention includes the use of other agents instead of or in combination with iodixanol.

[0058] FIG. 2 illustrates another set of operations collectively designated as **130** and which are directed to forming a layered array in a vessel, such as previously described array **10** and vessel **20** in FIG. 1. Preferably, a suitable vessel is obtained for subsequent receiving of the layered array. The vessel is preferably adapted for centrifugation and may be one specifically for use with the particular centrifuge used in operation **140** described below. The layered array is preferably formed by preparing one or more liquids for use as underlayer(s) in the vessel. These underlayers preferably comprise the density adjusting agent such as iodixanol and one or more other liquids such as water. The proportion of the density adjusting agent is selected such that the density of the underlayer is greater than that of the injection layer. For a process of length separation of SWCNTs in iodixanol and water, the underlayer preferably comprises at least 30% iodixanol. After formation of the liquid for use as the desired underlayer, the liquid is transferred into the vessel, such as by pipetting. After formation of the one or more underlayers, the injection layer containing the dispersed SWCNTs to be separated, is then deposited on the underlayer(s) in the vessel. After formation of the injection layer, the race layer is formed. As noted, the race layer comprises an amount of the density adjusting agent, such as iodixanol, and one or more additional liquids such as water. The proportion of the density adjusting agent, e.g. iodixanol, is selected so that the density of the race layer(s) is less than that of the injection layer. Preferably, for separating SWCNTs using iodixanol and water, the race layer typically comprises less than about 30% and preferably less

than about 25% iodixanol. The liquid for use as the race layer is then transferred into the vessel and deposited on the injection layer to thereby form a race layer. An upper layer may optionally be formed on top of the race layer(s).

[0059] The preferred process **100** further comprises a centrifugation operation shown in FIG. 2 as operation **140**. In this operation, the vessel containing the layered array is subjected to application of very large acceleration forces. Preferably, centrifugation is performed to create centrifugal speed forces of at least 10,000 g; preferably 25,000 g, and most preferably at least 40,000 g. Centrifugation is performed for a time period sufficient to enable a collection of fractions to form within the vessel. Typically, multiple layered fractions will form in the region of the vessel occupied by the race layer(s). As explained herein, the various respective fractions contain SWCNTs separated by length.

[0060] Various types of centrifuges can be used for operation **140**, such as for example fixed angle centrifuges, swinging bucket centrifuges, vertical centrifuges, and depending upon the application, near vertical tube rotors.

[0061] Selection of a rotor depends on a variety of conditions, such as sample volume, number of sample components to be separated, particle size, desired run time, desired quality of separation, type of separation, and the centrifuge in use. Fixed angle rotors are general purpose rotors that are especially useful for pelleting particles and in short-column banding. Tubes are retained at an angle (usually 20 to 45 degrees) to the axis of rotation, typically in numbered tube cavities. The tube angle shortens the particle pathlength, compared to swinging bucket rotors, resulting in reduced run times.

[0062] Swinging bucket rotors allow tubes in swing outward. Gradients of all shapes and steepness can be used.

[0063] Vertical tube rotors hold tubes parallel to the axis of rotation; therefore, bands separate across the diameter of the tube rather than down the length of the tube.

[0064] Near vertical tube rotors are designed for gradient centrifugation when there are components in a sample mixture that do not participate in the gradient. The reduced tube angle of these rotors significantly reduces run times from the more conventional fixed angle rotors, while allowing components that do not band under separation conditions to either pellet to the bottom or float to the top of the tube.

[0065] Selection of a suitable vessel for centrifuging the layered array also depends upon numerous factors such as, but not limited to the centrifugation technique to be used, including the rotor in use, volume of sample to be centrifuged, need for sterilization, importance of band visibility, and so forth; chemical resistance—the nature of the sample and any solvent or gradient media; temperature and speed considerations; and whether tubes or bottles are to be reused.

[0066] Informative guides as to the selection and use of centrifuges, rotors, tubes and accessories are provided by Beckman Coulter, under the designations “Centrifuges, Rotors, Tubes & Accessories, Ultracentrifuges,” publication BR-8101L; and, “Rotors and Tubes, for Beckman Coulter Preparative Ultracentrifuges, User’s Manual,” publication LR-IM-23.

[0067] Without wishing to be bound to any particular theory that may limit the present invention, the following is presented to more fully describe the behavior of populations of dispersed carbon nanotubes of varying lengths, and how they react when subjected to various forces that result in their separation by length.